भारतीय मानक
Indian Standard

IS 14499 : 2021

रंग रोगन के लिए स्ट्रोंशियम क्रोमेट — विशिष्टि

(पहला पुनरीक्षण)

Strontium Chromate for Paints — Specification

(*First Revision*)

ICS 87.060.10

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Raw materials for paints, varnishes and related products Sectional Committee, CHD 21

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw materials for Paints, Varnishes and Related Products Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1998. Strontium chromate, SrCr04, is a yellow precipitated compound. It is non-bleeding, easily ground and has good light fastness as well as good heat resistance.

At present, it is mainly used in vinyl resin systems for fabric coatings and in metal-protective coatings. It mainly finds its use as a primer pigment for metal protective and corrosion resistance. It is used on Aluminium, Magnesium and their alloys and on vinyl sheeting.

Raw material for paint products may contain lead and other heavy and toxic metals either because of use as catalyst or their compounds being essential adjunct of formulation in manufacturing of paints. Taking cognizance of the fact that lead and toxic heavy metal exposure of human being, particularly children in residential premises is injurious to health and having adverse impact on human health and safety, the Committee responsible for formulation of this standard has felt the need to introduce restriction of lead and toxic heavy metal restriction in all paint raw material product standards. Revision of this standard has been taken up with a view to incorporate the maximum permissible limit of lead and heavy metal restriction clause in this standard. Therefore, in this revision, lead restriction up to a maximum permissible limit of 90 ppm and toxic heavy metals restriction (cumulative other than lead) as 0.1 percent maximum has been prescribed to avoid hazardous impact of lead and heavy metal exposure and consequent adverse impact on environment and human health. Further, majority of consumers are not aware of the consequences of lead and other heavy metal toxicity and its long-term implications to human health. Therefore, in this revision, along with lead and heavy metal restriction, a suitable cautionary notice has been included in the marking clause

The composition of the technical committee responsible for formulation of this standard is given in Annex K.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

STRONTIUM CHROMATE FOR PAINTS — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for strontium chromate, used for paints.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate by dated or undated the possibility of applying the most recent editions of the standards:

IS No	*Title*
33 : 1992	Inorganic pigments and extenders for paints — Methods of sampling and test (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
1303 : 1983	Glossary of terms relating to paints (*second revision*)
101 (Part 2/Sec 2) : 1986	Methods of sampling and test for paints, varnishes and related products: Part 2 Test on liquid paints (chemical examination), Section 2 Volatile matter (*third revision*)
101 (Part 8/ Sec 5) : 1993	Methods of Sampling and test for paints, varnishes, and related products: Part 8 Tests for pigments and other solids, Section 5 Lead restriction test
460 (Part 2) : 2020	Test sieves — Specification: Part 2 Perforated plate test sieves (*fourth revision*)

3 TERMINOLOGY

For the purpose of this standard, the terms and definitions given in IS 1303 shall apply.

4 REQUIREMENTS

4.1 Composition

The pigment shall be commercially pure strontium chromate free of extenders and organic material for materials introduced to improve those properties for which the pigment is used.

4.2 The mass colour and character of the tint and tinting strength formed by a mixture with a white pigment shall be as agreed upon between the purchaser and the supplier when tested in accordance method prescribed in Annex A.

4.2.1 The material shall also conform to the requirements and given in Table 1.

4.3 Lead Restriction

The material shall be tested for restriction from lead in accordance with IS 101 (Part 8/Sec 5), or by Atomic Absorption Spectrometer (AAS) or by ICP. When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead exceeding 90 ppm (*see* Note).

NOTE — When no lead is used during production (lead free).

4.4 Toxic heavy Metal Restriction

Product shall not be manufactured using mercury and mercury compounds, cadmium, chromium VI, arsenic, antimony and their oxides. The material shall not contain more than 0.1 percent by weight in total of above toxic heavy metals in the form of natural impurities or impurities entailed by the production process which are contained in the raw material, when tested by the relevant atomic absorption spectroscopic methods or by ICP method.

Table 1 Requirements for Strontium Chromate for Paints

(*Clause* 4.2.1)

Sl No.	Characteristic	Requirements	Method of Test Ref to Annex
(1)	(2)	(3)	(4)
i)	Relative density. g/ml	3.67 to 3.82	B
ii)	Oil absorption value, g/100g	Shall be within ± 10 percent of the oil absorption value of the approved sample.	C
iii)	Strontium as SrO , percent by mass, *Min*	47	D
iv)	Chromium as CrO_3, percent by mass, *Min*	44	E
v)	Chloride as Cl, percent by mass, *Max*	0. I	F
vi)	Nitrate as NO_3, percent by mass, *Max*	0. I	G
vii)	Sulphate as SO_3 percent by mass, *Max*	0.2	H
viii)	Total residue on 45 microns IS sieve, percent by mass, *Max*	0.5	J
ix)	Volatile matter, percent by mass, *Max*	0.2	IS I01 (Part 2/Sec 2) : 1986

5 PACKING AND MARKING

5.1 Packing

The material shall be suitably packed as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 The containers shall be marked with the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Lead content (maximum);
d) Toxic heavy metals content;
e) Net mass of the material;
f) Batch number or lot number in code or other-wise;
g) Month and year of manufacture;
h) Cautionary note as below:
  1) Keep out of reach of children, and
  2) This product may be harmful is swallowed or inhaled.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act,* 2016 and the Rules and Regulations 2018 framed there under, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed under IS 33.

7 TESTS

7.1 Tests shall be conducted as referred in co1 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 4.2)

DETERMINATION OF COLOUR AND STRENGTH

A-1 OUTLINE

A-1.1 This method is intended to be used to compare the colour and strength of a pigment under test with a reference standard of the same type and grade.

A-1.1.1 The method does not apply to white pigments.

A-1.2 Pigments are dispersed in a suitable vehicle with a mechanical muller. Test and standard pigments are treated identically. Opaque drawdowns are made from the dispersions and compared, either visually or instrumentally, for colour and strength differences.

A-2 APPARATUS

A-2.1 Balance — Sensitive to 1.0 mg.

A-2.2 Mechanical Muller — Equipped with ground-glass plates to which a variable but known force may be added in 220-N increments. The driven glass plate shall have a speed of rotation of between 70 and 120 rpm and the apparatus shall have an arrangement for pre-setting the number of revolutions in multiples of 50.

A-2.3 Rubbing Surfaces — The rubbing surfaces of the ground glass plates shall be kept sharp by removing them from the muller and grinding them face-to-face with No. 303 optical emery, or its equivalent, and water.

A-2.4 Small Glass Slab — Or other nonabsorbent material, suitable for weighing and mixing pigment pastes.

A-2.5 Spatula — Having 75 to 150 mm blade.

A-2.6 Paper Charts — White with a black band, with surface impervious to paint liquids.

A-2.7 Film Applicator — At least 75 mm wide with a clearance of 100 mm to produce wet films about 50 mm thick.

A-2.8 Colour-Measuring Instrument

A-3 MATERIALS

A-3.1 Reference Standard

A standard pigment of the same nature as the pigment to be tested, as agreed upon between the purchaser and the supplier.

A-3.2 Vehicle

A solvent-free vehicle, such as No. 1 lithographic varnish, with0.8 percent each of cobalt and manganese driers (6 percent) types.

A-3.3 White Tinting Paste

A white tinting paint compatible with the dispersion vehicle, such as 57 parts of rutile titanium dioxide dispersed in 43 parts of the vehicle described in **A-3.2**.

NOTE — Because the choice of vehicle and white tinting pigment may affect the results, these should be agreed upon between the purchaser and the supplier.

A-4 PROCEDURE

A-4.1 Decide, by agreement or by experimentation the following dispersing conditions:

a) the force applied to the muller plates;
b) the number of revolutions;
c) the mass of the pigment; and
d) the mass of the vehicle.

A-.4.2 Applying these decisions, prepare a dispersion of the reference standard pigment. Weigh onto a glass slab, to within 2 mg, the appropriate quantities of the standard pigment and the dispersing vehicle. Mix the pigment and vehicle together thoroughly with the spatula and transfer the mixture to the lower plate of the muller. Spread the mixture in a path approximately 100 mm wide and halfway between the center and rim of the lower plate, and clean the spatula as much as possible by wiping it on the upper plate of the muller. Close the plates and carry out the mulling stages of SO revolutions; after each stage collect the paste from both plates with the spatula and spread it around the 100 mm path on the lower plate, wiping the spatula on the upper plate as before. When the mulling has been carried out for the prescribed number of revolutions, collect the paste, and store it. Clean the glass slab, the Muller plates, and the spatula, and repeat the procedure with exactly the same quantities of the test sample and vehicle. Collect the paste from this sample and store it.

NOTE — The most common sources of error in this procedure are inaccurate weighing, incomplete transfer of the pigment and vehicle mixture, and contamination of the plates by previous samples.

A-4.3 Tint Colour

A-4.3.1 Determine by calculation the amount of white pigment paste that must be added to 0.5 g of the colour pigment paste so that the mixture contains 1 part of dry colour pigment to 10 parts of dry white pigment. For stronger or weaker pigments this ratio may be adjusted accordingly, for example, 1 : 20 or 1 : 5 respectively.

A-4.3.2 Weigh 0.500 + 0.002 g of the standard colour pigment paste onto a glass slab. Then weigh the amount of white pigment paste. Determined in **A-4.3.1**, and place it next to the colour pigment paste on the glass slab. Thoroughly mix the two pastes together with the spatula until a uniform colour is observed.

A-4.4 Calculation of Tinting Strength

If the colours of the test tint paste and the standard tint paste are visually the same. The tinting strength of the test pigment is equal to that of the standard pigment, and the relative tinting strength of the test pigment is 100 percent. However, if the test and standard colours are not the same, the difference may be due to tinting strength. To determine the relative tinting strength of the test pigment, repeat the operations of **A-4.3.2**, but this time use an amount of the test pigment paste that is estimated to give the closest colour match to the standard pigment paste. Repeat this procedure until satisfied that the closest colour match has been obtained. At this point any residual colour difference between the test and the standard pigments is attributed to a shade difference, rather than a strength difference. Note and record this shade difference. Calculate the relative tinting strength of the test pigment by dividing the mass of the standard paste by the mass of the test paste used to obtain the closest colour match; multiply by 100 to express the result in percent.

ANNEX B

[*Table* 1, *Sl No*. (i)]

DETERMINATION OF RELATIVE DENSITY

B-0 PRINCIPLE

The method described here applies to solids only and consists in finding out the mass of water occupying the same volume as a known mass of the sample. Then the relative density of the sample under test is the ratio of mass of the sample to mass of water occupying the same volume as the sample.

B-1 PROCEDURE

Weigh accurately a clean and dry Regnault's specific gravity bottle/volumetric flask of 50 ml capacity (W_1). Take about 5 g of the sample into the bottle/ flask and weigh again (W_2). Add about 30-35 ml water containing 0.5 percent of a suitable wetting agent (for example, Calsolene oil HS), washing down any portion of sample sticking to the inside wall of the bottle. Shake thoroughly with the stopper in place till all the sample is thoroughly wetted. Wait for IS minutes or until such time when all the air bubbles are eliminated. Add more of water containing 0.5 percent of wetting agent to make up to the mark and weigh (W_3). Pour out the contents of the bottle and wash thoroughly with water. Finally fill up the bottle to the mark with distilled water and weigh (W_4). Make a note of the temperature at which the determination is carried out.

NOTE — The density of water remains practically unchanged on addition of 0.5 percent of its own mass of wetting agent. Hence this value does not significantly differ from the actual.

B-2 CALCULATION

$$Relative\ Density = \frac{W_2 - W_1}{(W_4 - W_1) - (W_3 + W_1)}$$

ANNEX C

[*Table* 1, *Sl No*. (ii)]

DETERMINATION OF OIL ABSORPTION

C-0 PRINCIPLE

The oil absorption of a pigment or extender, is the mass of oil absorbed by a given mass of pigment or extender under closely defined condition. It is usually expressed as a percent on the mass taken.

C-1 APPARATUS

C-1.1 Glass Plate, 300 mm × 300 mm × 10 mm upper side ground.

C-1.2 Palette Knife, 250 mm × 25 mm.

C-2 REAGENTS

C-2.1 Acid Refined, linseed oil of acid value 7.5 to 8.5.

C-3 PROCEDURE

Weigh out a quantity of the sample in accordance with the expected oil absorption (*see* Note). Place the pigment on the surface of the glass plate, add 3 or 4 drops of oil from the burette, and rub it into the pigment with the palette knife. Continue adding small quantities of oil, rubbing after each addition until stiff paste of smooth consistency is obtained. This paste should just spread without cracking or crumbling. Read the burette reading and note the quantity of oil used. The time taken for the complete operation must be between 20 to 25 min. Allow the paste to stand for one or two minutes and rub again to see that no appreciable change has taken place.

C-4 CALCULATION

NOTE

Expected oil absorption	Mass of pigment to be taken in g
15	20
16-30	10
31-50	5
51-80	2
81 and above	1

ANNEX D

[*Table* 1, *Sl No*. (iii)]

DETERMINATION OF STRONTIUM

D-1 REAGENTS

D-1.1 Acetic Acid (Glacial)

D-1.2 Ammonium Hydroxide (1 : 3)

Mix 1 volume of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) with 3 volumes of water.

D-1.3 Ethyl Alcohol

D-1.4 Hydrochloric Acid (1 : 1)

Mix equal volume of concentrated hydrochloric acid (sp gr 1.19) and water.

D-1.5 Sulphuric Acid (1 : 19)

Mix one volume of concentrated sulphuric acid (H_2S04, sp gr 1.84) with 19 volumes of water.

D-2 PROCEDURE

D-2.1 Weigh to the nearest mg about 0.5 g of the sample into a 400 ml beaker. Add 40 ml of HCI (1 : 1) and heat the solution to dissolve the pigment. Add 40 ml of ethyl alcohol until the chromium is reduced, as this is indicated by a dark colouration of the solution. Add to the solution 100 ml of water and sufficient NH_4OH (1 : 3) to form a slight persistent precipitate.

D-2.2 Add HCl drop wise until the precipitate just re-dissolves (*see* Note). Heat the solution to just under boiling and add 20 ml of H2S04 (1 : 19). Add 100 ml of ethyl alcohol and allow the precipitate to settle for several hours or overnight.

NOTE — For a cleaner precipitate, add HCl until pH of 2.0 is reached, instead of adding HCI drop-wise until the precipitate is just dissolved. Then add 25 ml of acetic acid to minimize the tendency of the Sulphate to form a complex with trivalent chromium. Finally, heat the solution and add H_2S04 (1 : 19).

D-2.3 Filter the precipitate through a Gooch crucible that has been previously dried to constant weight. Wash the precipitate several times with a solution of equal volumes of ethyl alcohol, water, and H_2S0_4 where (1 : 19). Dry the crucible in an oven and then ignite for 1/2 h at 800 °C or until constant mass is attained. Cool and weigh the crucible.

D-3 CALCULATION

D-3.1 Calculate the percent of strontium oxide (SrO) as follows:

$$SrO, Percent\ by\ mass = [(P \times (0.56416)/S] \times 100$$

where

P = g of SrS04 precipitate, and

S = g of sample pigment.

ANNEX E

[*Table* 1, *Sl No*. (iv)]

DETERMINATION OF CHROMIUM

E-1 REAGENTS

E-1.1 Hydrochloric Acid (1 : 5)

Mix 1 volume of concentrated hydrochloric acid (HCL, sp gr 1.19) with 5 volumes of water.

E-1.2 Potassium Iodide Solution

Dissolve I50 g of potassium iodide (KI) in water and dilute to 1 litre.

E-1.3 Sodium Thiosulphate Standard Solution (0.1 N)

Dissolve 24.8 g of reagent grade $Na_2S_2O_3.5H_20$ in freshly boiled water and dilute to 1 litre with additional freshly boiled water. To standardize, pipette 25 ml of the standard potassium dichromate solution (exactly 0.100 0 N) into a 250 ml Erlenmeyer flask. Add 100 ml of water, 15 ml of potassium iodide solution, and 15 ml of concentrated hydrochloric acid (sp gr 1.19). Titrate the liberated iodine with sodium thiosulphate solution until the reddish-brown colour becomes quite faint. Add 5 ml of starch solution and continue the titration drop wise until the blue colour changes to a pale green. Calculate the normality of the sodium thiosulphate solution as follows:

$$N = \frac{2.5000}{M}$$

where

N = normality; and

M = millilitres of sodium thiosulphate solution.

E-1.4 Starch Indicator Solution

Make a homogenous paste of 10 g of soluble starch in cold water. Add to this 1 litre of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/litre) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to 10 °C. Prepare fresh indicator when the end point of the titration, from blue to colour less or blue to light green, fails to be sharp.

E-1.5 Weigh accurately approximately 0.2 g of the sample into a 250 ml iodine flask. Add 50 ml of water and 50 ml of HCI (1 : 5); swirl the flask to complete solution. Add 20 ml of KI solution and allow the solution to stand for several minutes.

E-1.6 Titrate the liberated iodine with 0.1 N $Na_2S_20_3$ solution until the reddish brown iodine colour becomes faint. Add 1 ml of starch solution and continue the titration cautiously to the end point which is easily discernible when the colour changes from blue to a light green with no blue tinge.

E-2 CALCULATION

Calculate the percent of chromium as chromic oxide (CrO3) as follows

$$CrO_3, Percent\ by\ mass = [(V\,N \times (0.0334)/S] \times 100$$

where

V = millilitres of $Na_2S_20_3$ solution required for titration of specimen,

N = normality of $Na_2S_20_3$ solution, and

S = g of sample used.

ANNEX F

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF CHLORIDE CONTENT

F-1 REAGENTS

F-1.1 Silver Nitrate Solution

Dissolve 17.0 g of silver nitrate in water and dilute to 1 litre.

F-2 PROCEDURE

F-2.1 Weigh 10 g of the sample into a beaker, add 200 ml of water and 50 ml of nitric acid. Warm just enough to dissolve the sample. Filter to remove the insolubles. Add a slight excess of silver nitrate. Boil for 5 minutes and let it stand for 2 h.

F-2.2 Filter at room temperature through a tared glass crucible. Wash the precipitate free of silver nitrate with 1 : 99 nitric acid. Dry the precipitate at 105 ± 2 °C for 2 h, cool and weigh as silver chloride.

F-3 CALCULATION

Calculate the percent of chlorides as chlorine as follows:

$$\textit{Chlorine,percent}\ (M \times 0.247)/m \times 100$$

where

M = the mass of precipitate, in g; and

m = the mass of sample used.

ANNEX G

[*Table* 1, *Sl No.* (vi)]

G-1 DETERMINATION OF NITRATE CONTENT – METHOD A

G-1.1 Reagents

G-1.1.1 *Hydrochloric Acid* (sp gr 1.18).

G-1.1.2 *Sodium Hydroxide Solution* (200 g/l).

G-1.1.3 *Ammonium Chloride Solution* (17.2 mg/l).

G-1.1.4 *Devarda's Alloy* (powdered).

G-1.1.5 *Ammonia-Free Water*

Redistill approximately 500 ml of distilled water to which has been added 1 g of anhydrous sodium carbonate and 1 g of potassium permanganate. Reject the first 100 ml of distillate and then collect about 300 ml.

G-1.1.6 *Nessler's Reagent*

Prepare by either of the methods detailed below in **G-1.1.6.1** or in **G-1.1.6.2**.

G-1.1.6.1 Dissolve 5 g of potassium iodide in 3.5 ml of water. Add cold saturated mercury (II) chloride ($HgCl_2$) solution, while stirring, until a faint red precipitate is formed. With continued stirring add 40 ml of 50 percent potassium hydroxide solution, dilute to 100 ml with water, mix well, allow to settle, decant the clear supernatant liquid and store it in the dark.

G-1.1.6.2 Dissolve 3.5 g of potassium iodide and 1.25 g of mercury (II) chloride in 80 ml of water. Add cold saturated mercury (II) chloride solution, while stirring, until a slight red precipitate remains, then add 12 g of sodium hydroxide, stir until dissolved, and finally add a little more saturated mercury (II) chloride solution and dilute to 100 ml with water. Stir occasionally during several days allow to stand and use the clear supernatant liquid for the test.

G-1.2 Apparatus

G-1.2.1 *Distillation Apparatus*

G-1.2.2 *Nessler Cylinders* - 50 ml capacity.

G-1.2.3 *Mechanical Agitator or Stirrer*

G- 1.3 Procedure

G-1.3.1 Weigh 30 ± 0.1 g of sample in a glass flask. Agitate this sample with 300 ml of water by mechanical agitator or stirrer for 1 h at room temperature in such a manner that the pigment is kept in continuous suspension without increasing the temperature of the water. Filter the mixture and reserve the clear filtrate for testing.

G-1.3.2 Take 50 ml of above filtrate into the distillation flask and dilute to 150 ml with ammonia-free water. Add 3 g of Devarda's alloy and 30 ml of sodium hydroxide solution and close the apparatus at once. Place 2 ml of hydrochloric acid and 30 ml of ammonia-free water in the receiver. Warm the flask gently until the reaction starts and then allow the reaction to proceed gently for about half an hour. Then distil about 70 ml of liquid, the receiver being kept cool with running water. Make up the distillate to 250 ml with ammonia-free water and transfer 5 ml to a Nessler cylinder. Dilute to 50 ml with ammonia-free water. Transfer 5 ml of ammonium chloride solution (equivalent to 0.1 percent $N0_3$) into a similar Nessler cylinder and dilute to 50 ml with ammonia-free water. Add 1 ml of Nessler's reagent to each cylinder and mix each thoroughly. Allow both cylinders to stand for 5 min and compare the intensity of colour of the two solutions.

G-1.4 Expression of Results

Report the result as either greater than or less than 0.1 percent $N0_3$.

G-2 DETERMINATION OF NITRATE CONTENT-METHOD B

G-2.1 Principle

The nitrate present in the test sample is used to nitrate salicylic acid in sulphuric acid medium. The nitro-compound formed is of an intense yellow colour in alkaline solution, and the colour is measured spectrophotometrically at a wavelength of 410 nm.

G-2.2 Principle

G-2.2.1 *Sulphuric Acid* (sp gr 1.84).

G-2.2.2 *Sulphuric Acid* (5N).

G-2.2.3 *Ethanol 95 Percent* (*v*/*v*).

G-2.2.4 *Sodium Salicylate Solution* (5 g/l, freshly prepared).

G-2.2.5 *Sodium Hydroxide Solution* (300 g/l).

G-2.2.6 *Sodium Hydroxide Solution* (4N).

G-2.2.7 *Potassium Nitrate* (dried at 120 °C and cooled in a desiccator).

G-2.3 Apparatus

G-2.3.1 *Spectrophotometer* — Suitable for measurements at a wavelength of 410 nm.

G-2.3.2 *10 mm Cells* — For use with the spectrophotometer.

G-2.3.3 *pH Meter*

G-2.3.4 *Volumetric Flasks* — Of capacity 50 ml, 100 ml, 250 ml and 500 ml.

G-2.4 Preparation of Calibration Graph

G-2.4.1 *Standard Solution I* — Weigh 163 ± 0. 1 mg of potassium nitrate, dissolve it in water in a 100 ml volumetric flask, make up to the mark and mix well.

G-2.4.2 *Standard Solution II* — Dilute 10 ml of standard solution I to a volume of 500 ml.

G-2.4.3 Measure 2, 4, 6, 8 and 10 ml of standard solution II (corresponding to 0.04, 0.08, 0.12, 0.16 and 0.2 mg of NO_3 respectively) into separate 100 ml glass beakers.

G-2.4.4 *Treat Each Beaker as Follows*

Add 1 ml of sodium salicylate solution, evaporate to dryness on a water bath and allow to cool in a desiccator. Moisten the whole of the dried residue with 1 ml of sulphuric acid and allow to stand in the desiccator for 10 min. Afterwards, wash the contents into 50 ml volumetric flasks with water, add 10 ml of sodium hydroxide solution and cool to room temperature. Make up to the mark with water and mix well. Determine and record the optical density at 410 nm in a 10 mm cell against a water blank.

G-2.4.5 Construct a graph of optical density against milligrams of $N0_3$.

G-2.5 Procedure

G-2.5.1 Take 50 ml of filtrate mentioned in **G-1.3.1** above, into a 250 ml glass beaker and add 5 ml of sulphuric acid and 2 ml of ethanol. Warm the solution until the chromate is reduced as indicated by a change of colour to green. Boil the solution vigorously to drive off organic matter, taking care to avoid losses by splashing, cool and add sodium hydroxide solution until just alkaline. Cool again and adjust the *p*H to 8.0 + 0.5 measured by the *p*H meter. Filter through filter paper and wash with hot water, collecting the filtrate and washings. Cool, make up the volume to 250 ml and mix.

G-2.5.2 Transfer 10 ml (*see* Note) of this solution to a 100 ml glass beaker. Add 1 ml of sodium salicylate solution and proceed as described in **G-2.4** including the determination of the optical density at 410 nm.

G-2.5.3 From the known optical density of the test solution, determine from the calibration graph the corresponding mass of nitrate in milligrams.

NOTE — If the nitrate content is more than 0.1 percent, carry out a second determination, using 5 ml of solution.

G-2.6 Expression of Results

Calculate the nitrate content as a percentage by mass, expressed as NO_3 by the formula m, when 10 ml of solution have been used; and 2 m, when 5 ml of solution have been used (*see* Note to **G-2.5**); where m is the mass, in milligrams, of NO_3 corresponding to the optical density of the test solution.

ANNEX H

[*Table* 1 *Sl. No*. (vii)]

DETERMINATION OF SULPHATE CONTENT

H-1 REAGENTS

H-1.1 Barium Chloride Solution

Dissolve 117.0 g of barium chloride in water and dilute to 1 litre.

H-2 PROCEDURE

H-2.1 Weigh about 20 g of pigment into a litre beaker. Add 75 ml of hydrochloric acid (sp gr I. 19). Boil the solution under a hood until the rapid evaluation of chlorine has subsided. Add 5 ml of methyl alcohol and boil to a volume of about 35 ml (*see* Note). Add 25 ml of hydrochloric acid and continue boiling. Add 5 ml of methyl alcohol and again boil to a volume of about 35 ml.

H-2.2 Dilute the solution to 400 ml with water. If the solution is not clear at this point, filter it. Heat to boiling and add 50 ml of glacial acetic acid. Add 10 ml of barium chloride solution, drop wise, while stirring. Boil for 30 min and allow to stand overnight. Filter through a tared Gooch crucible and wash with hot water. Dry the precipitate in an oven and ignite at 900 °C to a constant mass. Weigh as barium sulphate.

H-3 CALCULATION

Calculate the percent of sulphate as SO_3 as follows:

$$SO_3\textit{, percent} = (\mathrm{M} \times 0.0343)/m \times 100$$

where

M = mass of precipitate, in g; and

m = mass of sample used.

ANNEX J

[*Table* 1, *Sl No*. (viii)]

DETERMINATION OF TOTAL RESIDUE

J-1 PROCEDURE

J-1.1 Dry the sieve (45 microns (*see* IS 460 Part 2) in an oven at 105 ± 2 °C; cool, and then weigh to the nearest 1 mg.

J-1.2 Weigh accurately 10 g of material. Wet the sieve on both sides with alcohol and transfer the material to the sieve and wet with alcohol.

J-1.3 Hold the sieve under a tap delivering about 300 to 500 ml of the wash liquid (water) per minute. By slightly shaking the sieve, the pigment will be rapidly carried through. A soft camel's hair brush may be used in aiding the operation. If the sieve is held at a slight angle so that the pigment gradually collects at one edge during the washing process, and then rotated, the pigment may be brushed out rapidly, with no risk of clogging the sieve.

J-1.4 After most of the finely divided portion of the pigment has passed through the sieve place the sieve in a porcelain dish containing 250 ml of the wash liquid so that the sieve is covered to a depth of about 15 mm. Brush the pigment remaining on the sieve with a soft, 25 mm camel's hair brush at the rate of 2 strokes per second during 2 periods of 10 s each. Rinse the sieve from the dish after each period to let the liquid run through. Change the liquid in the dish after every 2 brushing periods. Continue this operation until the wash liquid passing over the residue and through the sieve is clear and free from solid particles. When the washing appears to be complete, collect about 200 ml of the wash liquid in a clean beaker. Stir the liquid vigorously and set the beaker on a black surface. The washing is not considered complete until such a test fails to show any particles collected about the middle of the bottom of the beaker.

J-1.5 When the washing is complete wash the pigment particles adhering to the brush back on to the sieve and wipe off the water below the sieve. Add a few drops of alcohol and then of ether to expedite drying. Dry the sieve for one hour at 105 °C; cool and weigh to constant mass. Calculate the percent residue on sieve.

ANNEX K

(*Foreword*)

COMMITTEE COMPOSITION

Raw Materials for Paints, Varnishes and Related Products Sectional Committee, CHD 21

Organization	*Representative(s)*
In personal capacity (Retired from IIT Bombay)	Prof A. S. Khanna (***Chairman***)
Office of Micro, Small and Medium Enterprises, New Delhi	Shri Sunil Kumar Newar Shri Santosh Kumar (*Alternate*)
Harcourt Butler Technical University, Kanpur	Dr P K Kamani Dr Arun Maithani (*Alternate*)
ICT, Mumbai	Prof Sabnis Prof Mahanwar (*Alternate*)
Indian Institute of Chem. Technology, Hyderabad	Dr Ramanuj Narayen Dr Pratyay Basak (*Alternate*)
Institute of Minerals and Materials Technology	Nominationa Awaited
University Institute of Chemical Technogy, Jalgaon	Prof Ravindra Puri
Shriram Institute of Industrial Research	Shri A. K. Majumdar
Central Electro Chemical Research Institute, Karaikudi	C. Arunchandran D. Sherwood
National Test House, Ghaziabad	Nomination Awaited
Naval Materials Research Laboratory	Nomination Awaited
Birla White	Dr R K Singh Shri Om Prakash Gautam (*Alternate*)
Asian Paints Ltd, Mumbai	Mr Rajeev Kumar Goel Dr Subrahmanya Shreepathi (*Alternate*)
Atul Limited	Dr Mahesh Soni Dr Jayesh Joshi updated nominees (*Alternate*)
Berger Paints India Limited, Kolkata	Shri T. K. Dhar Shri Swagata Chakraborty (*Alternate*)
Clariant Chemicals (India) Ltd, New Delhi	Shri Nitin Vaidya Shri Umesh Kapoor (*Alternate*)
Heubach India, Mumbai	Shri J. I. Sevak
Hindustan Zinc Limited, Delhi	Shrimati Payal Chauhan
Indian Chemical Council	Nomination Awaited
Indian Lead Zinc Development Association, New Delhi	Shri K. Sridhar
Indian Small Scale Paint Association	Shri Mukund Hulyalkar Shri Mukesh Goyal (*Alternate*)
J J Chemicals	Nomination Awaited
Kansai Nerolac Paints Ltd, Mumbai	Shri Laxman Nikam Shri Manoj Kumar Somani (*Alternate*)
Shalimar Paints Limited	Shri Gokul C. Sutradhar Shri Chinmaya Nayak (*Alternate*)

Organization	*Representative(s)*
SSPC India Chapter	Dr Buddhadeb Duari Subhajit Bhattacharya Kaushik Duari (*Young Professionals*)
20 Microns Limited, Mumbai	Dr Anil Bansal
Rubamin Limited	Nomination Awaited
Vapi Oxide Colours Pvt Ltd (VOXCO), Vapi	Shri Ishan Raveshia Shrimati Minal Mayekar (*Alternate*)
Consumer Voice	Shri M. A. U. Khan Shri B. K. Mukhopadhaya (*Alternate*)
Director General, BIS	Ajay K. Lal Scientist 'E' and Head (CHD) [Representing Director General (*Ex-officio*)]

Member Secretary

Pushpendra Kumar
Scientist 'B' (CHD), BIS

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